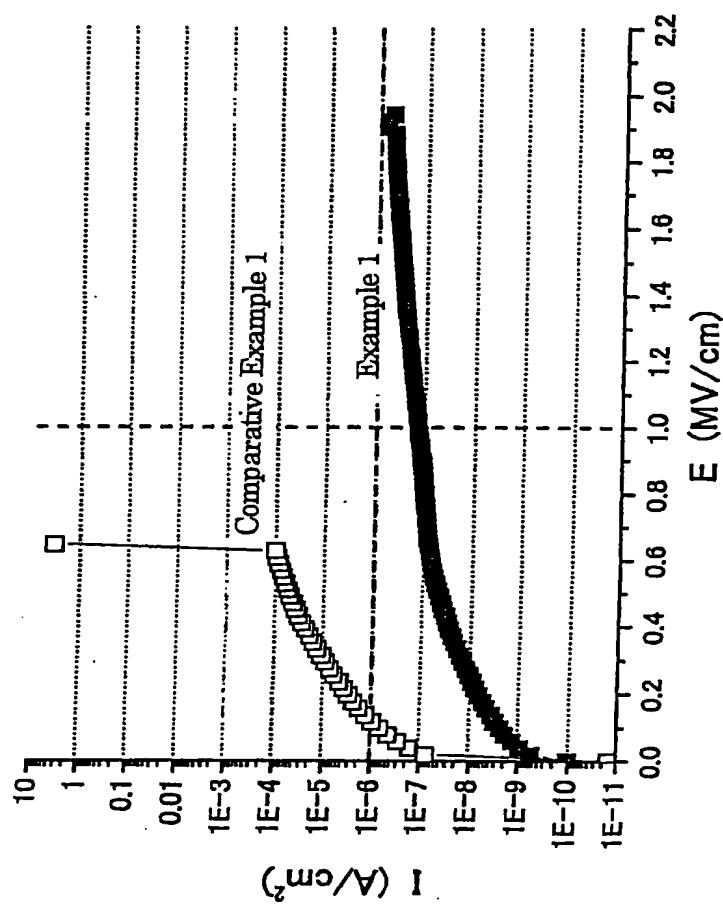
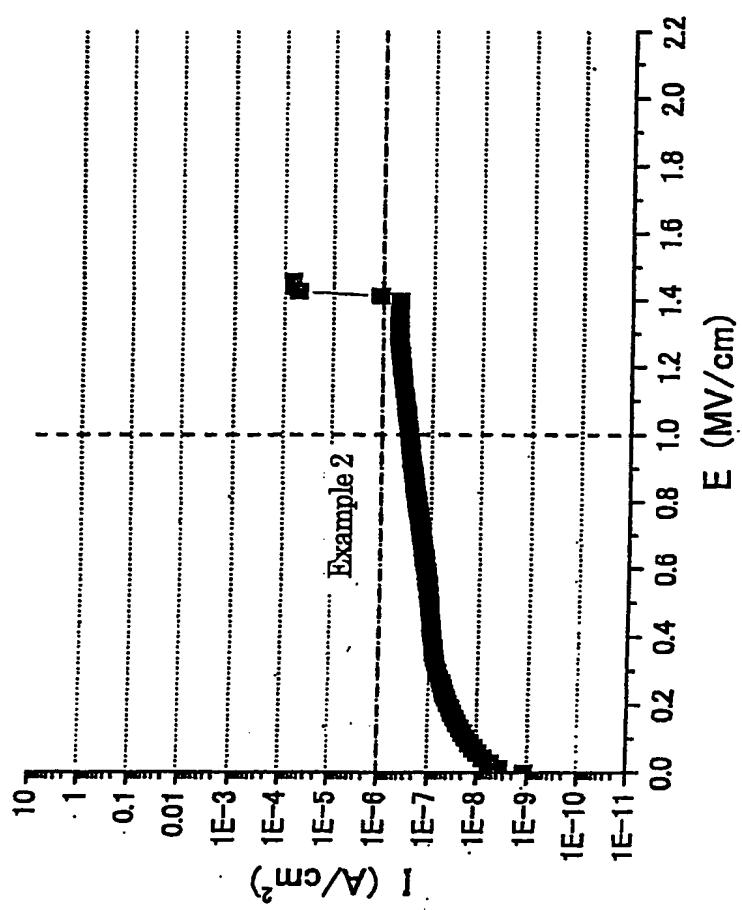


[Fig. 1]



[Fig. 2]



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**[Document Name] Claims**

**(Claim 1)**

A low dielectric constant film comprising a film comprising at least fine diamond particles and pores, wherein the surface of said fine diamond particles is treated with at least one single substance of the following (a) or a mixture of substances of at least one of (a) and at least one of (b) as described below:

(a) a substance represented by the general formula:



(b) a substance represented by the general formula;



(where n = 1 or 2, m = an integer of 0 to 3, X represents a halogen group, C<sub>1</sub> to C<sub>6</sub> alkoxy group or phenoxy group, and R represents C<sub>1</sub> to C<sub>6</sub> alkyl group).

**(Claim 2)**

The low dielectric constant film of claim 1, wherein X is at least one member selected from the group consisting of chlorine group, methoxy group and ethoxy group and R represents methyl group or ethyl group in the general formula (a) and the general formula (b).

**(Claim 3)**

The low dielectric constant film of claim 1 or 2, wherein m is 1 in the general formula (a) and the general formula (b).

**(Claim 4)**

The low dielectric constant film of any one of claims 1 to 3, wherein n is 1 in the general formula (a).

**(Claim 5)**

The low dielectric constant film of claim 1, wherein said substance of the general formula (a) is at least one member selected from the group consisting of dichloro tetramethyl disiloxane, dimethoxy tetramethyl disiloxane, tetrachloro dimethyl disiloxane and tetramethoxy dimethyl disiloxane, and said substance of the general formula (b) is at least one member selected from the group consisting of hexachlorodisiloxane, hexamethoxy disiloxane and hexaethoxy disiloxane.

**(Claim 6)**

A low dielectric constant film comprising a film comprising at least fine

diamond particles and pores, wherein the surface of said fine diamond particles is treated with at least one member of the general formula (b) in which X represents a C<sub>1</sub> to C<sub>6</sub> alkoxy group or phenoxy group.

(Claim 7)

An electronic part comprising the low dielectric constant film of any one of claims 1 to 6 as at least one constituent element.

(Claim 8)

A method of manufacturing a low dielectric constant film, said method comprising the step of chemically reacting hydroxyl groups on the surface of fine diamond particles and at least one single substance of (a) or a mixed substances of at least one of (a) and at least one of (b) described above.

(Claim 9)

The method of manufacturing a low dielectric constant film of claim 8, wherein said substance of the general formula (a) is at least one member selected from the group consisting of dichloro tetramethyl disiloxane, dimethoxy tetramethyl disiloxane, tetrachloro dimethyl disiloxane and tetramethoxy dimethyl disiloxane, and therein said substance of the general formula (b) is at least one member selected from the group consisting of hexachlordisiloxane, hexamethoxy disiloxane and hexaethoxy disiloxane.

**[Document name] SPECIFICATION**

**[Name of the Invention] LOW DIELECTRIC CONSTANT FILMS OF DIAMOND FINE PARTICLES, ELECTRONIC PARTS AND MANUFACTURING METHOD THEREOF**

**[TECHNICAL FIELD]**

**[0001]**

The present invention concerns a low dielectric constant thin film as an insulating film of a porous structure in which fine diamond particles are bonded, and a manufacturing method thereof, as well as an electronic part such as a semiconductor integrated circuit device of high integration degree and high speed operation type using the same.

**[BACKGROUND ART]**

**[0002]**

Among semiconductor integrated circuit devices, particularly in super LSI devices, delay of signals passing through wirings prepared in devices gives rise to a significant problem along with lowering of power consumption as the wirings become finer and are more integrated. Particularly, in a high speed logic device, RC delay due to the resistance and the distribution capacity of wirings results in most prominent subject and, among all, it is necessary to use a material of low dielectric constant for inter-wiring insulating materials in order to decrease the distribution capacity.

**[0003]**

Heretofore, as an insulating film in a semiconductor integrated circuit, a silica film ( $\text{SiO}_2$ ), a tantalum oxide film ( $\text{Ta}_2\text{O}_5$ ), an aluminum oxide film ( $\text{Al}_2\text{O}_3$ ), a nitride film ( $\text{Si}_3\text{N}_4$ ) and the like have been used and, particularly, as an insulating material between multilayer wiring, a nitride film and a silica film doped with an organic material or fluorine have been used or studied as the low dielectric constant film. Further, as an insulating film for further lowering the dielectric constant, a fluororesin, a silica film formed by baking a foaming organic silica film, a porous silica film formed by depositing fine silica particles, etc. have been studied.

**[0004]**

Until now, since diamond is more excellent in the heat conductivity and the mechanical strength than other materials, this is a material suitable to heat dissipation for semiconductor devices of high integration degree and large heat generation amount

and has been studied in recent years. For example, JP-A No. 6-97671 proposes a diamond film of 5  $\mu\text{m}$  thickness by a film forming method such as a sputtering method, ion plating method or cluster ion beam method. Further, JP-A No. 9-263488 proposes a film forming method of scattering fine diamond particles on a substrate and growing diamond crystals using them as nuclei by supplying carbon by a CVD (Chemical Vapor Deposition) method.

[0005]

The present inventors have obtained a specific dielectric constant of 2.72 by a fine diamond particle film of a porous structure as already disclosed in JP-A No. 2002-110870. Further, since fine diamond particles do not bond to each other, this results in a problem that the film strength is lowered and, in order to solve the problem, JP-A No. 2002- 89604 proposes a reinforcing method by crosslinking fine diamond particles by means of a hexachlorodisiloxane treatment and it is shown that a specific dielectric constant comparable with that in JP-A No. 2002-110870 is obtained also by the treatment. Further, the present inventors have reported that a specific dielectric constant of 2.1 is obtained by heating and purifying fine diamond particles in a mixed acid comprising sulfuric acid/nitric acid in the Academic Conference.

[0006]

【Patent Document 1】 JP-A No. 6-97671

【Patent Document 2】 JP-A No. 9-263488

【Patent Document 3】 JP-A No. 2002-110870

【Patent Document 4】 JP-A No. 2002-289604

【Non-Patent Document 1】 The 50th meeting of the Japan Society of Applied Physics and Related Societies, Pre-text No. 2, p 913 (2003).

[0007]

Materials known so far as having low dielectric constant are listed in the following table.

Name of material	Specific dielectric constant
Silica (plasma CVD)	4.2 - 5.0
Fluorine-added silica	3.7
Diamond (single crystal)	5.68
Porous silica	1.5 - 2.5
Porous diamond	2.1 - 2.72
Polyimide	3.0 - 3.5
Polytetrafluoroethylene	1.9
Gas	1

**[DISCLOSURE OF THE INVENTION]**

**[Problems to be solved by the Invention]**

[0008]

As described above, for further improvement of the integration degree, various studies have been made for obtaining materials having a specific dielectric constant being lower than 3.7 of the fluorine-added silica in the table. Since the silica film per se comprises two kinds of atoms elements of oxygen and silicon of high electronegativity, orientation polarization remains and it is insufficient as the low dielectric constant film, so that porous silica produced by a blowing method of fine particles has been studied. However, they are insufficient in the mechanical strength and have not yet been put to practical use. Further, while polytetrafluoroethylene as the fluororesin shown in the table has a sufficient specific dielectric constant, this can not be used since a severe condition for the required heat resistance from 300°C to 400°C or higher in the semiconductor manufacturing process can not be satisfied. While the polyimide is a heat resistant resin, it is carbonized at 300 to 400°C or higher and can not be used as well.

[0009]

The inventors has successfully obtained a low dielectric film having a

mechanical strength and a sufficient specific dielectric constant, by bridging and reinforcing fine diamond particles with hexachlorodisiloxane.

[0010]

It has been found in a further study that electric characteristics were insufficient in which the dielectric breakdown voltage was 0.58 MV/cm while it should be 1.0 MV/cm or higher and the leak current value as the reciprocal of the insulation resistance was  $10^{-4}$  A/cm<sup>2</sup> at 0.58 MV/cm while it should be  $10^{-6}$  A/cm<sup>2</sup> or less as shown by the symbol “□” in Fig. 1.

**[Effect of the Invention]**

[0011]

Further, in the fine porous diamond particle film treated according to the invention has been improved by 100 times or more and the dielectric breakdown voltage has also been improved by 3.5 times or more than those in usual, to reach a practical level. Further, the specific dielectric constant which is a most prominent feature of the fine porous diamond particle film is unchanged as 2.1 before and after the treatment or lowered to about 1.9 and the function as the low dielectric constant film is sufficiently maintained. Further in a case where sulfonic groups or carboxy groups are present on the surface of the fine diamond particles, further lowering of leak current and increase of the dielectric breakdown voltage could be obtained by combination with the treatment with the metal salt such as barium chloride. Further, diamond is known as a material of good heat conductivity, and the heat conductivity is not deteriorated even when pores are formed compared with the existent SOG film.

**[Means for Solving the Problems]**

[0012]

As a result of seeking for the cause by using infrared absorption spectroscopy, it was found that when purified fine diamond particles are treated with hexachlorodisiloxane, a broad absorption spectrum for hydroxy group increased at the wave number of 3400 cm<sup>-1</sup>. It is considered that the hydroxy groups form HO-Si bonds due to the hydrolysis of unreacted Cl-Si bond of hexachlorodisiloxane with water content in air. In the course of the study described above, the present inventors have

found that the leak current was caused and the dielectric breakdown voltage is not increased by the water molecules having affinity with the hydroxy groups and impurities contained in a trace amount.

[0013]

The present inventors have made an earnest study on the reinforcing agent for the fine diamond particle film with an aim of improving the electric characteristics and, as a result, have developed a low dielectric constant film having at least fine diamond particles and pores, characterized by a reinforcing treatment of crosslinking fine diamond particles to each other, by treating the surface of the fine diamond particles with at least one member of the substances at least represented by the following general formula (a), thereby obtaining a remarkable improvement.

(a) General formula:  $X_nR_3 - nSi(OSi)mR_3 - nX_n$

(in which  $n = 1$  or  $2$ ,  $m =$  an integer of  $0$  to  $3$ ,  $X$  represents halogen group,  $C_1$  to  $C_6$

alkoxy group or phenoxy group, and  $R$  represents  $C_1$  to  $C_6$  alkyl group).

[0014]

Further, a low dielectric constant film having a sufficient strength and satisfactory electric characteristics could be obtained by treating the surface of the fine diamond particles not only with the substance represented by the above general formula (a) alone, but also with mixed substances of at least one of the substances represented by the following general formula (b) and the substance represented by (a) described above.

(b) Substance represented by general formula:  $X_3Si(OSi)mX_3$

(where  $m =$  integer of  $0$  to  $3$ ,  $X$  represents halogen group,  $C_1$  to  $C_6$  alkoxy group or phenoxy group).

The method of treating the fine diamond particle film includes, for example, a method of dipping in a liquid containing at least the compound of the general formula (a) and/or (b) (hereinafter referred to as a reinforcing agent), a method of coating a liquid containing the reinforcing agent on the film, a method of spraying a liquid containing the substance used in the invention on the film, or a method of treatment by exposing

the film to the reinforcing agent as alone or being diluted with a solvent as vapor under heating or at an ambient temperature.

[0015]

In a case of mixing the compound of the general formula (a) and the compound of the general formula (b), while (a) and (b) may be from 2:98 to 98:2 by weight ratio, it is more preferred that they are at 70:30 to 10:90. The weight ratio is based on a mixed weight ratio in a liquid state before treating the surface of the fine diamond particles. In a case of treatment in a gas state, it is necessary to take the vapor pressure of each substance at a treating temperature into consideration. Hexachlorodisiloxane has higher reactivity than hexamethoxydisiloxane to the hydroxy group. However, the former releases hydrogen chloride and the latter releases methanol as a by-product. In a case of forming the low dielectric constant film of the invention in the course of manufacturing a semiconductor circuit, since chlorine ions may sometimes give undesired effects, the latter is preferred as the treating agent. However, in a circumstance where chlorine ions are removed sufficiently, the former is used. Accordingly, in a case of using the former and the latter in admixture, the mixing ratio can be determined optionally in accordance with the situation of the processes for manufacturing various semiconductor circuits.

[0016]

In the above general formula (a) and/or (b), X is at least one halogen group selected from the group consisting of fluorine, chlorine, bromine, and iodine, or alkoxy group having C<sub>1</sub> to C<sub>6</sub> alkyl or phenoxy group, R is C<sub>1</sub> to C<sub>6</sub> alkyl group. In a case where C is more than 6 both for the alkoxy group and the alkyl group, the reaction rate with the hydroxy group is lowered, which is not preferred. Further, the phenoxy group (C<sub>6</sub>) has reactivity and can be used in the invention. The criterion for selecting either the halogen group or the alkoxy group as X can be determined in view of the above-described reactivity and the chlorine ion removing circumstance.

[0017]

Then, in the above general formula (a) and/or (b), in a case where m exceeds 3, the siloxane chain is lengthened to increase the viscosity, thereby making it difficult for impregnation between fine diamond particles in a case of treatment in the liquid state, which is not preferred. Further, also in a case of treatment with vapors of the siloxane compound, the boiling point is increased when m exceeds 3, which is not preferred.

Accordingly, a case where m is 1, that is, a disiloxane compound is most preferred.

[0018]

In the above general formula (a) and/or (b), it is most preferred that X is at least one member selected from the group consisting of reactive chlorine group, methoxy group or ethoxy group and R is a hydrophobic methyl group or ethyl group. In addition, it is preferred that m is 1, i.e., a disiloxane compound and further that n is 1 or 2, i.e., two or four chlorine groups or alkoxy groups such as methoxy groups or ethoxy groups or four or two methyl groups or ethyl groups.

[0019]

The fine diamond particle film having pores of the invention is formed on a semiconductor substrate such as a single crystal or polycrystal silicon substrate, a compound semiconductor substrate, a quartz substrate, a ceramic substrate, and a glass substrate, or on an intermediate product substrate for manufacturing semiconductor formed with various circuits. A colloidal solution of the fine diamond particle is coated on the surface of the substrate after the hydrophilic treatment by oxidation.

[0020]

In addition to the treatment for the film per se deposited by coating the colloidal solution of the fine diamond particles as described above with the compound of the above general formula (a) and/or (b) of the invention, it may be sometimes necessary to improve the adhesion between the substrate and the film. In this case, the adhesion between the substrate and the fine diamond particle film can be improved also by applying a pretreatment with the compound of the above general formula (a) and/or (b), among all, one or mixture of hexachlorodisiloxane and hexamethoxydisiloxane between the hydrophilic treatment and the coating. In this case, treatment such as drying and heating may optionally be applied for sufficient reaction.

[0021]

In the invention, in a case where X is C<sub>1</sub> to C<sub>6</sub> alkoxy group or phenoxy group in the general formula (b): X<sub>3</sub>Si(OSi)<sub>m</sub>X<sub>3</sub>, it may be used as a treating agent without mixing with the substance (a). This substance can react with hydroxy groups on the surface of fine diamond particles at a room temperature or by heating to conduct crosslinking between the fine particles.

[0022]

In a case where hydroxy groups or silanol groups such as the hexachlorodisiloxane residue remain on the surface of the fine diamond particles after reinforcement formed on the substrate, they may be made hydrophobic by treating with hexamethyl disilazane, monomethoxy silane, monochloro silane, etc.

[0023]

Since sulfuric acid or nitric acid is sometimes used in the purifying step for the fine diamond particles used in the invention, sulfonic groups or carboxy groups may be formed on the surface. In this case, at least one metal ions of the group consisting of calcium, strontium, barium, mercury, silver, lead or radium may be bonded to the groups. The method of treating the low dielectric constant film with the metal ions includes, for example, a method of forming a fine diamond particle film having pores, then selecting a water soluble salt such as a hydride, hydrogen chloride or nitrate of the metal and dissolving the metal in water, impregnating the solution into the pores of the fine diamond particle film and bonding the same with the carboxy groups and/or sulfonic groups bonded to the surface of the fine diamond particles, or adding the solution of the metal salt to the dispersion liquid of the fine diamond particles. In this case, for removing unnecessary metal salts, sufficient water washing is applied followed by drying. In a case of conducting the metal salt treatment and the fine particle bonding treatment together, any of them may be conducted previously but conduction of the former previously is easy for treatment.

[0024]

Further, combination of the metal salt treatment and the hydrophobic agent treatment with hexamethyl disilazane or the like described above provides the effect of both of them, thereby further improving the insulation resistance and dielectric breakdown voltage.

[0025]

The fine diamond particles used in the invention are preferably solid particles with the particle size of from 1 nm to 1000 nm and purified to a purity of 95% or higher. Further, the porosity of the low dielectric constant film of the invention is preferably from 40% to 80%. In a case where it is 40% or less, the dielectric constant increases to 3 or more mainly in a case where the distribution of the particle size of the fine diamond particles is broad, which is not preferred. Further, in a case where it is 80% or more, no mechanical strength can be obtained, which is insufficient for practical use.

[0026]

For preparing the colloid of the fine diamond particles, while an aqueous medium is generally used, it is preferred that the fine diamond particles in the dispersion medium are dispersed as primary particles of the particle size described above, but they can also be used when agglomerated to about 30 nm to 1000 nm in appearance to form secondary particles. For dispersion, a known fine particle dispersant or known viscosity modifier may also be used within a range not deteriorating the physical properties such as the dielectric constant, the electric resistance value, and the dielectric breakdown voltage.

[0027]

Since the fine diamond particle film of the invention has pores, the surface is naturally rough and, accordingly, it is preferably densified. For this purpose, a known method such as an SOG (Spin on Glass) method, an SG (Silicate Glass) film method, a BPSG (Boron Phosphate SG) film method or a plasma CVD method, or a method of coating a dispersion liquid of fine diamond particles of 5 nm or less, etc. can be used.

[0028]

The temperature for treating the deposited film formed by coating the colloidal solution of the fine diamond particles with the compound containing at least the compound of the above general formula (a) and/or (b) is conducted in a range from a room temperature to 400°C. While depending on the boiling point of the solvent used for diluting the compound, it is preferably treated at a temperature between about 50°C to 150°C and reacted simultaneously. Further, the deposited film of the fine diamond particles may be treated at a room temperature with a vapor or liquid containing the compound and then heated and reacted at a temperature between about 40°C and 400°C, preferably between about 50°C and 150°C.

[0029]

The invention also includes an electronic part having the low dielectric constant film described above as a constituent element. As the electronic part, a multilayer wiring type semiconductor integrated circuit of high integration degree and high speed operation type is most suitable but it may also be a usual semiconductor device or micro machine, or a usual capacitor having a low dielectric constant film of the invention comprising fine diamond particles and pores (fine porous diamond particle film).

## 【BEST MODES FOR CARRYING OUT THE INVENTION】

[0030]

Examples of the present invention are to be described below but the invention is not limited only to the examples.

### 【Example 1】

[0031]

#### <Preparation of colloidal solution>

In pure water in a beaker made of quartz, 5% by weight of purified fine diamond particles, 0.1% by weight of dimethylamine, and 1% by weight of polyethylene glycol with a molecular weight of 5,000,000 were charged and the beaker was dipped in an ultrasonic wave tank and sufficiently dispersed for one hour to obtain a gray viscous dispersion liquid.

#### <Spin coating step>

A thoroughly cleaned silicon substrate was cut in about 20 mm square, applied with a hydrophilic surface treatment, and then placed on a spindle of a spin coater, the colloidal solution described above was poured downward and the substrate was rotated at 1500 rpm to uniformly coat the same by a centrifugal force.

#### <Drying step>

The silicon substrate coated with a liquid of fine diamond particles was air-dried to form a film and then placed on a hot plate at 300°C and dried for one hour.

#### <Reinforcing treatment for film structure>

The silicon substrate with the fine diamond particle film was placed in a tightly closed vessel, put to inter-particle permeating sufficiently by exposure to a vapor of 10% dichlorotetramethyl disiloxane (DCTMDS) dissolved in dichloromethane at an ambient temperature for one hour, and then further applied with an overheating treatment at 300°C for one hour.

#### <Measurement of current-voltage characteristics>

A mercury electrode was placed on a film in an atmospheric air and a voltage was applied between it and the silicon substrate to measure the voltage, the current value, and the dielectric breakdown voltage, which were divided with a previously measured film thickness to calculate the electrolysis intensity.

[0032]

Fig. 1 shows the current-voltage characteristics of the film at 510 nm thickness obtained in Example 1. In the fine porous diamond particle film applied with a DCTMDS treatment, the dielectric breakdown voltage was improved by 3.5 times or more from 0.57 MV/cm to 2.0 MV/cm (measuring limit) compared with the film obtained in Comparative Example 1 to be described later. The leak current representing the insulation resistance was lowered from  $1 \times 10^{-4}$  A/cm<sup>2</sup> to  $7 \times 10^{-8}$  A/cm<sup>2</sup> at a voltage of 0.57 MV/cm and lowered to  $1 \times 10^{-7}$  A/cm<sup>2</sup> at 1 MV/cm when compared in the same manner. Further, the dielectric constant was unchanged as 2.0 before and after the treatment. As shown by the broken line in Fig. 3, while 1 MV/cm or higher of the dielectric breakdown voltage and  $1 \times 10^{-6}$  A/cm<sup>2</sup> or less of the leak current are generally required, both of them are cleared in the invention, to reach a practical level.

Further, the fine porous diamond particle film in this example had no disadvantages such as destruction of inter-particle bond and maintained sufficient strength upon measurement by contacting the electric characteristic measuring probe or upon finger touch friction.

#### **[Example 2]**

[0033]

Experiment was conducted in the same procedures except for treatment with a gas of a liquid mixture of 1% by weight of DCTMDS and 1% by weight of hexachloro disiloxane instead of 10% DCTMDS in Example 1. As a result of measurement, the film thickness was 680 nm and the specific dielectric constant was 2.1. A dielectric breakdown voltage of 1.43 MV/cm which was higher than 1 MV/cm and a leak current of  $2 \times 10^{-7}$  A/cm<sup>2</sup> which was less than  $1 \times 10^{-6}$  A/cm<sup>2</sup> at a voltage of 1 MV/cm were attained. Further, the fine porous diamond particle film of the example showed no disadvantages such as destruction of inter-particle bond and maintained sufficient strength even upon finger touch friction.

#### **[Comparative Example 1]**

[0034]

Experiment was conducted in the same procedures except for treatment with a gas of 1% by weight of a hexachlorodisiloxane solution instead of 10% by weight of

DCTMDS in Example 1. The film thickness was 510 nm. The fine porous diamond particle film had a dielectric breakdown voltage of 0.6 MV/cm and a leak current of  $1 \times 10^{-4}$  A/cm<sup>2</sup> just before and could not meet the requirement for 1 MV/cm or higher of the dielectric breakdown voltage and  $1 \times 10^{-6}$  A/cm<sup>2</sup> or less of the leak current as the practical standard.

**[Industrial Applicability]**

[0035]

According to the invention, the specific dielectric constant of 2.0 has been attained by using a fine porous diamond particle film which is an inorganic low dielectric constant film having high heat resistance and heat conductivity. Further, also the dielectric breakdown voltage has attained 2.0 MV/cm (measuring limit) or higher exceeding 1 MV/cm which is the practical standard and also the leak current has attained  $1 \times 10^{-7}$  A/cm<sup>2</sup> at the practical voltage of 1 MV/cm. This enables to manufacture not only multi-layered wiring semiconductor devices or semiconductor capacitors, as well as other high performance general purpose electronic parts such as high performance capacitors and inter-wiring insulators, and can greatly contribute to the development of electronic industry including computers.

**[BRIEF DESCRIPTION OF THE DRAWING]**

[0036]

**[Fig. 1]**

a graph showing the current-voltage characteristics of fine porous diamond particle film of Example 1 of the invention (symbol: ■) and a low dielectric film of Comparative Example 1 (symbol: □).

**[Fig. 2]**

a graph showing the current-voltage characteristics of the a dielectric constant film obtained in Example 2 of a fine porous diamond particle film of the invention (symbol: ■)

**[Document Name] Figure**

**【Document Name】 ABSTRACT**

**【Abstract】**

**【Problems】** While a fine porous diamond particle film has been known as a high heat resistant and low dielectric constant film and also has high mechanical strength and heat conductivity, and is expected as an insulating film for multi-layered wirings in semiconductor integrated circuit devices, it is insufficient in current-voltage characteristic and has not yet been put into practical use.

**【Means for Solving Problems】**

Fine porous diamond particle film is treated with a reinforcing agent containing either or both of dichlorotetramethyldisiloxane and dimethoxytetramethyldisiloxane to perform the bridging reaction of the particles. The particles can be thus fixed, thereby attaining a dielectric breakdown voltage of 1MV/cm or more and a leak current of  $10^{-6}$  A/cm<sup>2</sup> or lower, which are practical standards.

**【Selected Figure】** Fig. 1